National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1085b

Wear-Metals in Lubricating Oil

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of methods and in the calibration of apparatus used in the analysis of engine lubricating oils and other materials of similar matrix for metal content. A unit of SRM 1085b consists of 10 ampoules: five 5 mL amber borosilicate ampoules, each containing approximately 1.2 g of a blend of 21 constituent elements in a base oil at a nominal concentration of 300 mg/kg; and five ampoules, each containing approximately 1.2 g of a matching base oil intended for use as an analytical blank and for matrix matching.

Certified Values: The certified values, expressed as mass fractions [1], for 14 elements are listed in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [2]. The certified values are based on measurements using two or more independent analytical methods (see Methods). All values are based on measurements using a sample mass of at least 250 mg.

Reference Values: The reference values, expressed as mass fractions, for seven elements are given in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. The reference values for arsenic and chlorine are included even though they were not included in the 21 elements blended to the nominal 300 mg/kg concentration.

Information Values: The information values, expressed as mass fractions, are provided in Table 3 for the concentration of boron and calcium. Information values for the mass fraction of elements in the blank base oil are given in Table 4. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value. Each information value was determined by a single method.

Expiration of Certification: The certification of this SRM is valid until **31 January 2006**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements were performed by R. Demiralp, W.R. Kelly, A.F. Marlow, J.R. Sieber, G.C. Turk, and L.L. Yu of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh and M.S. Levenson of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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Certificate Issue Date: 09 August 2000

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Preparation: The oil blend of 21 elements at a nominal concentration of 300 mg/kg and matching base oil were prepared by Conostan Division, Conoco Specialty Products, Inc. (Ponca City, OK). The oil blend was prepared by gravimetrically blending 21 assayed, single-element concentrates into a white mineral base oil. The single element concentrates were assayed by classical wet chemical methods of analysis. Ampoules of SRM 1085b were filled and sealed under inert gas at NIST.

Use and Storage: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 250 mg should be used. The blank base oil ampoules should be used for the preparation of analytical blanks. The mass fraction of elements in the blank base oil (see Table 4) is insignificant relative to that of the 300 mg/kg blend. The SRM should be stored at room temperature and protected from temperature extremes. Any unused portion of an opened ampoule cannot be stored for future use and should be disposed of properly.

Caution: Small glass chips may separate from the ampoule when opened. Care must be taken to avoid contamination of the SRM.

Table 1. Certified Mass Fractions for Elements in SRM 1085b

Element	Mass Fraction (mg/kg)		
Silver	304.6	±	8.9
Barium	300.1	±	2.4
Cadmium	302.9	±	5.1
Chromium	302.9	±	3.9
Copper	295.6	±	8.5
Magnesium	297.3	±	4.1
Manganese	300.7	±	2.0
Molybdenum	300.6	±	3.2
Sodium	305.2	±	7.0
Nickel	295.9	±	7.4
Lead	297.7	±	6.8
Tin	299.4	±	4.8
Vanadium	297.8	±	4.6
Zinc	296.8	±	6.8

The results are expressed as the certified value ± the expanded uncertainty. The expanded uncertainty is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, taken here to be $k = 2$. The uncertainty in the certified value includes a source for the uncertainties for each method and a source that accounts for possible biases among the methods. The latter source is derived from a Type B distribution [3] based on the method results. The overall procedure is described in Reference [4].

Table 2. Reference Mass Fractions for Elements in SRM 1085b

Element	Mass Fraction (mg/kg)		
Aluminum	300.4	±	9.3
Arsenic	51.3	±	6.7
Chlorine	57.6	±	9.5
Iron	301.2	±	5.0
Phosphorus	299.9	±	7.2
Silicon	300.2	±	5.0
Titanium	301.1	±	2.9

The reference value is equal to the mean of the results of the cited methods. The uncertainty in the reference value includes a source for the uncertainties for each method and a source that accounts for possible biases among the methods. The latter source is derived from a Type B distribution [3] based on the method results. The combined uncertainty is expanded by a coverage factor of 2. The overall procedure is described in Reference [4].

Table 3. Information Mass Fraction for Boron in SRM 1085b

Element	Mass Fraction (mg/kg)
Boron	300
Calcium	298

Table 4. Information Mass Fractions for Blank Base Oil in SRM 1085b

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Silver	< 0.2	Manganese	< 0.04
Aluminum	< 7	Molybdenum	< 0.02
Arsenic	< 0.02	Sodium	≤ 0.3
Barium	≤ 0.2	Nickel	< 1
Calcium	< 5	Phosphorus	< 5
Cadmium	< 0.002	Lead	< 0.03
Chlorine	≤ 0.7	Silicon	< 5
Chromium	< 0.07	Tin	< 8
Copper	< 0.09	Titanium	< 1
Iron	< 2	Vanadium	< 0.005
Magnesium	≤ 0.3	Zinc	< 0.15

Methods

Silver	INAA, XRF, GRAV PREP	Manganese	ICPMS, XRF, GRAV PREP
Aluminum	XRF, GRAV PREP	Molybdenum	INAA, XRF, GRAV PREP
Arsenic	INAA	Sodium	INAA, GRAV PREP
Boron	GRAV PREP	Nickel	ICPMS, XRF, GRAV PREP
Barium	ICPMS, XRF, GRAV PREP	Phosphorus	XRF, GRAV PREP
Calcium	XRF, GRAV PREP	Lead	ICPMS, GRAV PREP
Cadmium	INAA, GRAV PREP	Silicon	XRF, GRAV PREP
Chlorine	INAA	Tin	INAA, XRF, GRAV PREP
Chromium	INAA, ICPMS, XRF, GRAV PREP	Titanium	XRF, GRAV PREP
Copper	ICPMS, XRF, GRAV PREP	Vanadium	ICPMS, XRF, GRAV PREP
Iron	XRF, GRAV PREP	Zinc	INAA, ICPMS, XRF, GRAV PREP
Magnesium	ICPMS, XRF, GRAV PREP		

ICPMS	Inductively coupled plasma mass spectrometry at NIST
INAA	Instrumental neutron activation analysis at NIST
XRF	X-ray fluorescence spectrometry at NIST
GRAV PREP	Gravimetric preparation of the SRM from assayed concentrates. Performed by Conostan Division, Conoco Specialty Products, Inc.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (1994).

[2] May, W.E., Gills, T.E., Parris, R., Beck, II, C., Fassett, J.D., Gettings, R.J., Greenberg, R.D., Guenther, F.R., Kramer, G., MacDonald, B.S., and Wise, S., "Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements," NIST Special Publication 260-136, (1999).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 72-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); available at http://physics.nist.gov/Pubs/.

[4] Levenson, M.S., Banks, D.L., Eberhardt, K.R., Gill, L.M., Guthrie, W.F., Liu, H.K., Vangel, M.G., Yen, J.H., and Zhang N.F., "An ISO GUM Approach to Combining Results from Multiple Methods," *J. Res. Natl. Inst. Stand. Technol.*, in press.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.